TENTH QUARTERLY PROGRESS REPORT

FERRITE DEVELOPMENT
CONTRACT NO.: DA-36-039-sc-5449

GENERAL CERAMICS & STEATITE CORP.
KEASBEY, N.J.

TENTH QUARTERLY PROGRESS REPORT

CONTRACT FOR SERVICES, FACILITIES, AND MATERIALS REQUIRED TO INVESTIGATE AND STUDY MAGNETIC FERRITES

PERIOD COVERED: From July 1, 1953 to September 30, 1953

CONTRACT NO.: DA 36-039-sc-5449

DEPT. OF ARMY PROJECT NO.: 3-93-00-503; 3-99-04-042

SIGNAL CORPS PROJECT NO.: 32-2005-D, 29-194B

PLACED BY: Laboratory Office, Signal Corps Procurement Agency
Contracting Division, Fort Monmouth, New Jersey

CONTRACTORS: General Ceramics and Steatite Corporation
Keasbey, New Jersey

OBJECT: To develop ferromagnetic ferrites of different permeabilities, and low magnetic losses, and to investigate conditions favorable to their production.

REPORTED BY: Dr. E. Albers-Schoenberg, Supervisor
Ephraim Gelbard, Project Engineer

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100-200) is continued. Two bodies of the nickel-
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discussed.

B) The study of bodies of the system MgO-NiO-ZnO-
CuO-Fe$_2$O$_3$ has still not come to an end. Out of
a great variety of measurements, two materials
have been selected to show the effect of nickel-
oxide content upon the magnetic properties. All
significant measurements are shown.

C) A preliminary remark hints at the possibility of
combining temperature stable bodies and high Q
bodies into one and the same material.

D) The field strength at the conditions of the μc
test has been measured as a function of initial
permeability. It is found that all measurements
heretofore performed were at a field strength of
less than 0.45 milli-oersted which validates the
measurement technique.

PART II

A) The uniformity and reproducibility test of MF-254
"G" body has been completed. The results of the
G-2 preparation resemble those of the first part.
Some conclusions are drawn and some general rules
established.

PART I

Body Development and Measurements made during the Tenth Quarter
of the Contract
_____ DA Project No.: 3-93-00-503 - SC Project No.:32-2005-D _____

A) Temperature Stable Bodies of the Lower Permeability Range

The following investigation is, in some respect, a continuation of the previous work on oscillator slugs. Now, however, the objective of our experiments has been changed and has taken on a more general feature.

Up to the present time, it is known that temperature stability may be achieved in two ways:

1) a body of relatively high permeability in the dense-fired state can be brought down in temperature coefficient (simultaneously decreasing μ) by underfiring. The porous material represents a diluted state of magnetic matter with lower permeability.

2) a dense or almost dense fired body containing a small percentage of "impurities" or, in other words, of non-magnetic constituents, may also exhibit a somewhat reduced permeability in conjunction with a flattened out temperature curve of μ_o. An example of this type of material is the MF-90 "B" material.

In both cases, of course, the basic composition has to be appropriate inasmuch as a moderate temperature coefficient of μ_o is required right from the beginning. Nickel-zinc ferrites of relatively high nickel and relatively low zinc content lend themselves for the first of the two aforesaid types of material while, as the example of the MF-90-B shows, mixed 4 or 5 component bodies comply with the requirements of the second group.

Actually, both situations are special cases of the primary objective: i.e., to reduce the temperature coefficient by reducing the magnetic efficiency. Table XLIX summarizes the properties at several firings of two new materials for temperature stability.

The table explains the feature of the "stability through porosity" method although body MF-1832-C is not yet to be taken as the conclusion of the investigation. The initial permeability of 370 for the dense-fired body is reduced to about 175 in the underfired state. The temperature curves (toroidal measurement) are illustrated by Graph 167. Up to about 75°C temperature stability is very good. Between 75 and 125°C the temperature coefficient gradually reaches +0.03%/°C. Body MF-1829-C shows the same general principle, but in this case a

PART I

A) - Continued

relatively favorable temperature curve is associated with a
water absorption of 5-6%, indicating a too soft structure of
the material. However, it may be possible to increase the
density of this material while retaining good stability.

As regards the behaviour at various frequencies 1829
maintains its initial permeability and Q-factor reasonably well
up to 5 Mcs. μ_0 remains quite stable up to 15 Mcs. This is
in accordance with previous experiences that bodies of the
system $NiO-ZnO-Fe_2O_3$, when in an underfired state, are useful
up to frequencies of the $10^6 - 10^8$ range.

The next report will supplement these findings with
further work on refinement of the second class of temperature
stable bodies (dense MF-90-"B" type).

B) Bodies of the System $MgO-NiO-ZnO-CuO-Fe_2O_3$

The Ninth Quarterly Progress Report, April/June 1953 contains a brief statement on bodies of the above-named system where MgO, NiO and CuO are present in about the same weight percentage (4-5%).

The body referred to was MF 1459. In continuation of this investigation, several more variations have been made with the objective of determining the changes in magnetic properties which occur when small changes of composition are made. Each compositional variation was subjected to calcine temperature variations, but in order to simplify any conclusions that may be drawn, a series of compositions which used the same calcine $(MgO-ZnO-Fe_2O_3)$ fired at a temperature of 2260°F is reported here. The firing temperature of the test samples was 2380°F. Table L shows the chemical compositions of two ferrites as well as a summary of the important magnetic properties. All measurements were performed upon C-shaped cores having about a 2 sq. cm. cross section and a mean magnetic path length of about ˜7 cm. The R. F. measurements were made at a frequency of 0.55 M. Each magnetic circuit had two air gaps, but the adjacent surfaces of the two C-cores were well ground to minimize the effects of the gaps.

If the variations in properties are related to the nickel oxide content of the body (which has proven through experience to be one of the most important parameters upon the efficiency of bodies of low nickel and reasonable zinc content), then it is to be expected that the magnetic properties improve as the nickel oxide content increases.

Graph 168 shows the μo vs. temperature behaviour for these two materials on the same plot. The overall temperature behaviour of μo is better for MF 1480 than for MF 1459. Although the μo for MF 1480 is shown lower than that for MF 1459 (which has less nickle oxide) toroidal ring measurements have shown that μo is actually higher. These results are also supported by the other magnetic measurements which are shown in Table L. In overall efficiency, MF 1480 (with the higher nickel-oxide content) is equal or better than MF 1459.

It should be pointed out that MF 1480 is a reproduction of MF 1429 (reported upon in the Sixth Quarterly Progress Report July/Sept. 1952), but using raw materials of a lesser degree of purity. In respect to this variation, MF 1480 compares favorably to MF 1429.

Photos of hysteresis loops (60 cps) of a low nickel oxide content body (MF 1102 - "H-1"), a medium nickel oxide content body (MF 1459), and a high nickel oxide content body (MF 419"H") are shown in Graph 169. As far as can be seen from the shape of

B) Continued

the curve, these materials are quite similar. The actual magnetic
properties, however, vary more than the 60 cps hysteresis loops
indicate. Graph 170 shows the μo vs. temperature curves for
these same three ferrites. It is interesting to note that the
ferrite with the medium nickel oxide content has the best tempera-
ture behaviour, which is explained by a difference in the ZnO
content.

C) Preliminary Remark

Recent experiments have given evidence that bodies basically belonging to the system $NiO-ZnO-Fe_2O_3$ can be made that combine the following properties:

a) μo around 110

b) Q-factor, in the frequency range of 500 Kc to 2 Mc, of several hundred

c) a temperature coefficient of approximately $+0.1\%/^\circ C$ for the toroidal core

Further details will be given in later reports.

D) Measurement of Field Strength (H) at the Conditions of
the μo (1 Mc) Test

The purpose of this investigation was to again evaluate
the test procedure for toroidal measurements of μo and Q (at
1 Mc) using the General Radio 916-A Impedance Bridge in conjunc-
tion with a suitable signal generator and null detector. (See
First Quarterly Progress Report April/June 1949). The need for
this re-evaluation of the measuring techniques arises from the fact
that it is known that many ferrites (especially those of low
coercive force) have a rather narrow region where the μo is
independent of the magnetizing field strength. One objective
of this work was to try to plot a curve of H_{ac} vs. μo so that
for any particular measurement heretofore made or for future
measurements, the exact conditions of magnetizing field can be
found for a ferrite of any measured initial permeability.

Since most of the ferrite developmental work is carried
out on the F 108 ring (approximate dimensions: O. D. = 4.7 cm,
I. D. = 3.6 cm, H = 0.6 cm), the subsequent measurements were
performed on this size toroid. The actual toroidal dimensions
varied from material to material by as much as $\pm$ 15% (due to
shrinkage variations).

Measurements of 50 toroids were made; about 5 rings each
of all the commercially available "Ferramic" types so as to get
the complete range of possible μo values. The following measure-
ment (1 Mc) were made on all the rings:

1) X_L - inductive component of the torcidal impedance

2) R - resistive or loss component of the toroidal impedance

3) E peak-peak-voltage cross the toroidal impedance

4) The dimensions of the ring

Using the data of the preceding measurements, the field
strength can be computed. The following formulas show the
relationships:

1) $L = \dfrac{X_L}{2\pi f}$ where   L = toroidal inductance in micro-
henrys

X_L = bridge reactance reading divided
by frequency in megacycles

f = frequency in megacycles

D) Continued

2) $Lo = 0.0046 \ N^2 h \ log (10) \dfrac{OD}{ID}$

           where   Lo = toroidal inductance in micro-henrys (air core)

                      h, OD, ID = toroidal dimensions in cm

                      N = number of turns

3) $\mu o = \dfrac{L}{Lo}$

           where μo = initial permeability

(For a complete development of the validity of these above expressions, see First Quarterly Progress Report April/June 1949)

4) $Q = \dfrac{X_L}{R}$ where   Q = Q of toroidal inductance

                          R = bridge resistance reading (ohms)

Since the voltage across the toroidal inductance contains a resistive and reactive component, it is sometimes necessary to solve for these components in order to accurately find the field strength.

5) $\delta = \tan^{-1} \left(\dfrac{R}{X_L}\right) = \tan^{-1} \left(\dfrac{1}{Q}\right)$

                where δ = loss angle

                    (also $\tan \delta = \dfrac{1}{Q}$)

6) $\theta = \tan^{-1} \left(\dfrac{X_L}{R}\right) = \tan^{-1} (Q)$

                where θ = Phase angle

7) E reactive = $\dfrac{E_{peak\ to\ peak}}{\sqrt{2}}$ x $(\sin \theta)$
r.m.s.

           where   E reactive r.m.s. = r.m.s. active component of the total voltage appearing across the inductance

D) Continued

8) $B_{max} = \dfrac{E \text{ reactive} \qquad r.m.s.}{4.44 \ (f) \ (N) \ (A) \ (10^{-2})}$

where B_{max} = maximum value of sinusoidal flux density in core (gauss)

A = cross sectional area of the core in square cm.

f = frequency in Mcs.

9) $H_{max} = \dfrac{B_{max}}{\mu_0}$ where H_{max} = maximum value of the sinusoidal field strength in core (oersteds)

It can be seen that when the Q exceeds 10, the value of sin θ in (7) very nearly equals 1 so the reactive and resultant components are approximately equal.

The results of the measurements and calculations of the 50 rings shows that for μ_0 values up to 1000 (at least) the magnetizing field strength was always less than 0.45 milli-oersteds. Since this situation existed, no curve need be plotted for H_{ac} vs. μ_0. During the course of the measurements, it was also found that a reduction of the bridge input voltage (nominally 1 volt at 1 Mc) had no effect upon the measured μ_0 values. This indicates that all the μ_0 measurements are valid and within the range where the initial permeability is independent of field strength.

PART II

Body Development and Measurements made during the Third Quarter
of the Contract
DA Project No.: 3-99-04-042 - SC Project No.: 29-194-B

A) Investigation of Reproducibility and Uniformity of Ferrite
    Materials

From the second batch of G-body (preparation G-2), toroids
have been pressed fired and measured, and the results are commu-
nicated below.

Again three different grain sizes were available:

a) all through 20 mesh

b) through 20 mesh and on 80 mesh

c) all through 80 mesh

From these fractions, the third (all fine) has been set aside.
(Very fine powders, because of their high air content and diffi-
culty in pressing, are usually discarded in favor of heavier grain
size materials.)

Again three different pressures have been applied: 5 tons, 10
tons and 15 tons corresponding to 1040, 2080 and 3120 psi. The
number of cores in each series, however, has been increased to
50. From these a small number has been used for preliminary fir-
ing tests so that finally a total number of 263 cores has been
subjected to measurement and evaluated. The firing has been done
in a small tunnel kiln of 8-3/4 feet length (the same as for the
G-1 cores). The peak temperature was 2360°F. The appearance of
the cores is very good. They are dense and shiny and have no
deficiencies as to shape and surface. The evaluation of the μo
measurements results in a distribution curve Graph 171 similar to
that for the G-1 series.

Table LI gives a detailed account for each of these six dis-
tribution curves of μo. The table indicates that, for any par-
ticular pressure the dust-free granulation yields the better
results. It also indicates that for any particular particle dis-
tribution, the higher pressing pressure gives better uniformity.

There are small differences between the percentage figures
of the G-1 and G-2 experiment. It is believed that these can be
accounted for by the difference in the number of cores subjected
to each of the two test series. (13 to 15 for G-1; over 40 for
G-2). The reasoning is the following:

PART II - A) Continued

a) the variation through firing will be very small
   for so small a batch

b) on the other hand, one single core deviating
   from the majority disturbs unduly the overall
   picture and percentage figures.

Surely the greater numbers of tested cores in the case of
G-2 are more favorable from a statistical point of view, so
that the G-2 experiment comes closer to practical working
conditions.

With respect to absolute magnitudes of μo, the G-2 mix
is clearly superior. In the measurement of the G-2 rings,
the ambient temperature was closely checked and corrections
made (+1.3%/°C) when the temperature varied more than one-
half degree centigrade from the 27°C norm. For the G-2 series,
the average absolute magnitude of μo was about 10% higher
than that of the G-1 test. The Q values of all the rings of
the G-2 series were within about $\pm$ 5% and showed no discernable
trends as a function of either pressure or particle size.
Since the G-1 test showed that very good dimensional uniformity
could be achieved by controlling particle size and pressing
pressure, no dimensional evaluation has been made for the G-2
test.

The conclusions to be drawn from the whole uniformity
experiment (G-1 and G-2) may be condensed to the following
statements:

a) A medium grain size (relatively large particles in
   mixture with a variety of smaller ones but exclud-
   ing the very finest) is most suitable and offers
   optimum pressing conditions.

b) The pressure should be high. A high die pressure
   narrows the magnetic tolerances.

c) It is possible to keep almost 100% of the production
   within $\pm$ 5% tolerance, (μo and Q) and a tolerance of
   $\pm$ 2-1/2% still gives a very satisfactory yield.

d) The location of the peak of the distribution curve
   depends on the method of preparation and the firing
   temperature. Having once established the procedure
   reproducibility will not be in doubt.

e) Dimensional uniformity can be good if the particle
   sizes and pressing pressures are held constant.

PART III

RESEARCH PLANNED FOR NEXT QUARTER*

1) The investigation of temperature-stable bodies of the lower permeability range (approximately 100-200) will be carried on. Bodies of the B-type, although somewhat lower in permeability (around 70), are also still under consideration (they offer the advantage of lower porosity).

2) The investigation of bodies of the system MgO-NiO-ZnO-CuO-Fe_2O_3 will presumably come to its final conclusion.

3) High-Q temperature-stable bodies will be included in conjunction with (1) above.

4) The uniformity and reproducibility test will be continued. Next body to be studied will be M. F. 141-"I". Reversible permeability measurements will be started.

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* Nitrogen firing of manganese-zinc bodies, as was planned in the last Quarterly Report, has been delayed because of slow delivery of several items necessary for improved firing facilities.

MANHOURS SPENT ON CONTRACT FOR THE PERIOD OF

<u>July 1, 1953 to September 30, 1953</u>

NAME	TITLE	HOURS
Dr. E. Albers-Schoenberg	Supervisor	116
Ephraim Golbard	Project Engineer	168
Joseph Katz	Ceramist Engineer	4
Samuel Ortepio	Ceramist Engineer	136
Melvin Eisenberg	Electrical Engineer	20
James Callahan	Ceramist Engineer	455
Mary Hudson	Meas. Technician	314
Ruth Wainwright	Meas. Technician	298
Agnes Arias	Ceramic Technician	120
Elizabeth Wetzel	Ceramic Technician	7
Dr. K. Wetzel	Ceramist Engineer	91
John Kurtz	Technician	86
William Schirger	Technician	40
Peter Covezza	Technician	41
Anthony Koseski	Technician	105
Charles Purzas	Technician	95
R. Reseter	Technician	11
Sigmund Golion	Technician	160

TABLE XLIV.

PROPERTY CHART OF TWO MATERIALS FOR TEMPERATURE STABILITY OF INITIAL PERMEABILITY

BODY	OXIDE SYSTEM	FIRING TEMPERATURE OF 2380°F				FIRING TEMPERATURE OF 2180°F				FIRING TEMPERATURE OF 2160°F			
		μ_o (1Mc)	T.C.(μo) %/°C	Water Absorp.	Q (1Mc)	μ_o (1Mc)	T.C.(μo) %/°C	Water Absorp.	Q (1Mc)	μ_o (1Mc)	T.C.(μo) %/°C	Water Absorp.	Q (1Mc)
MF 1829-C	NiO-ZnO-Fe2O3	500	Un-stable	Dense	13	c				120-140	<+.03 (25-125°C)	5-6	75-80
MF 1832-C	NiO-ZnO-Fe2O3 (less NiO than 1829)	370	Un-stable	Dense	36	170-180	<+.02 2.5 - 2.8 (25-100°C)		63				

TABLE L

COMPOSITION AND MAGNETIC PROPERTIES* (at 25°C) OF A SERIES OF TWO BODIES
OF THE SYSTEM MgO-NiO-CuO-ZnO-Fe$_2$O$_3$

Body Number	Composition										Magnetic Properties				
	Weight Percent					Mol Percent					μ_0**	Q**	μ_{max}	B_s***	H_c
	NiO	MgO		ZnO	Fe$_2$O$_3$	NiO	MgO	CuO	ZnO	Fe$_2$O$_3$					
MF 1459	4.52	2.08	1.59	16.30	70.40	7.08	11.83	6.72	23.20	51.16	780	7.7	2400	2860	0.19
MF 1480	6.62	3.06	.26	16.30	69.70	10.40	8.88	6.31	23.39	50.99	585	9.9	3170	3220	0.19

*All measurements performed on well ground C-shaped cores.

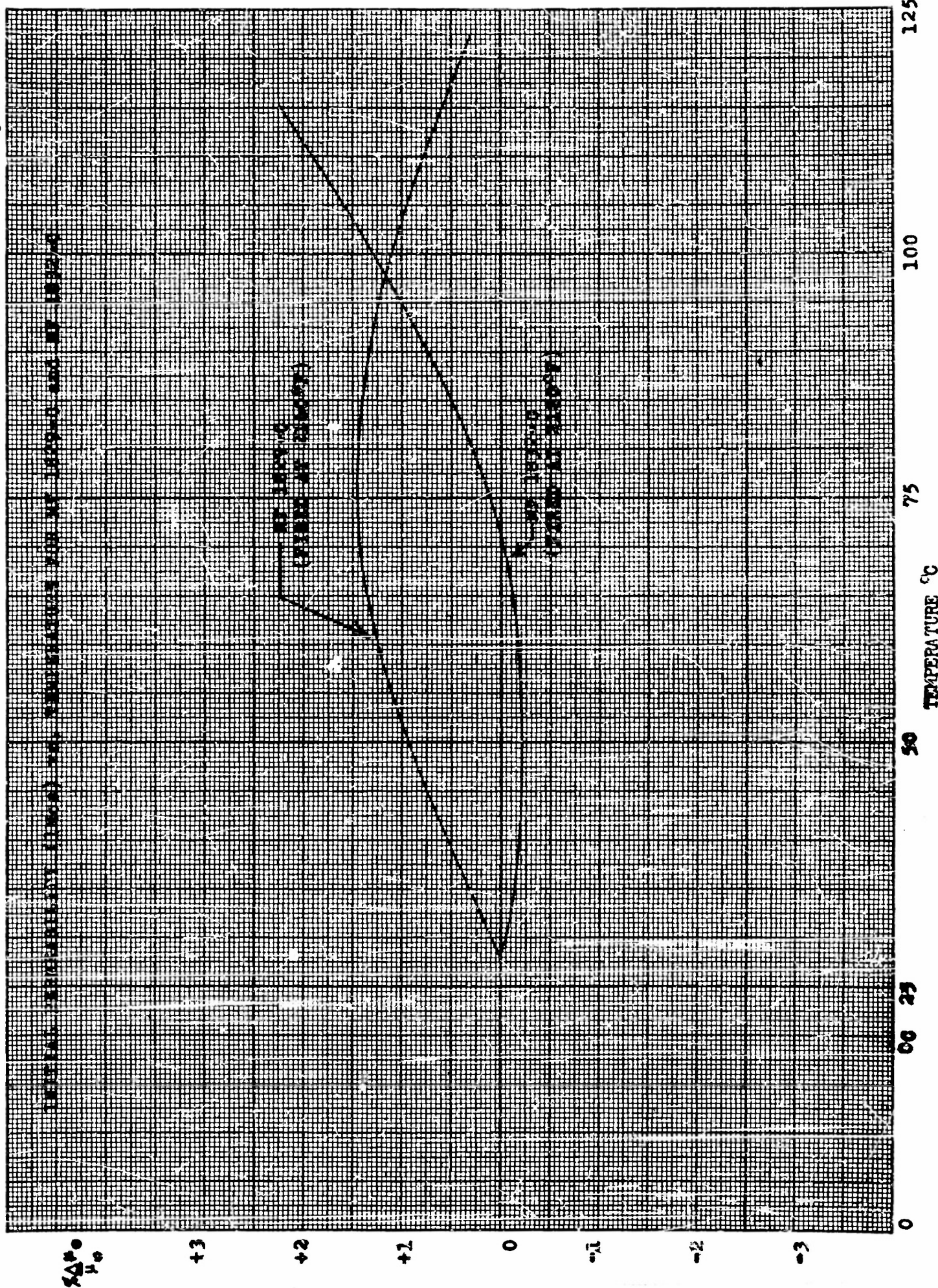** Measured at 0.55 °c

***At H$_{dc}$ = 27 Oersted

TABLE LI

DISTRIBUTION OF µo (1Mo) MF 254-"G" AT 27°C FOR THREE DIFFERENT
PRESSING PRESSURES AND TWO DIFFERENT PARTICLE SIZES (DIE F-109)
PREPARATION G-2 (PERCENTAGES OF CORES WITHIN ± 2-1/2% and ±5% FOR µo
(USING THE PEAK OF THE DISTRIBUTION CURVES AS THE CENTER VALUE)

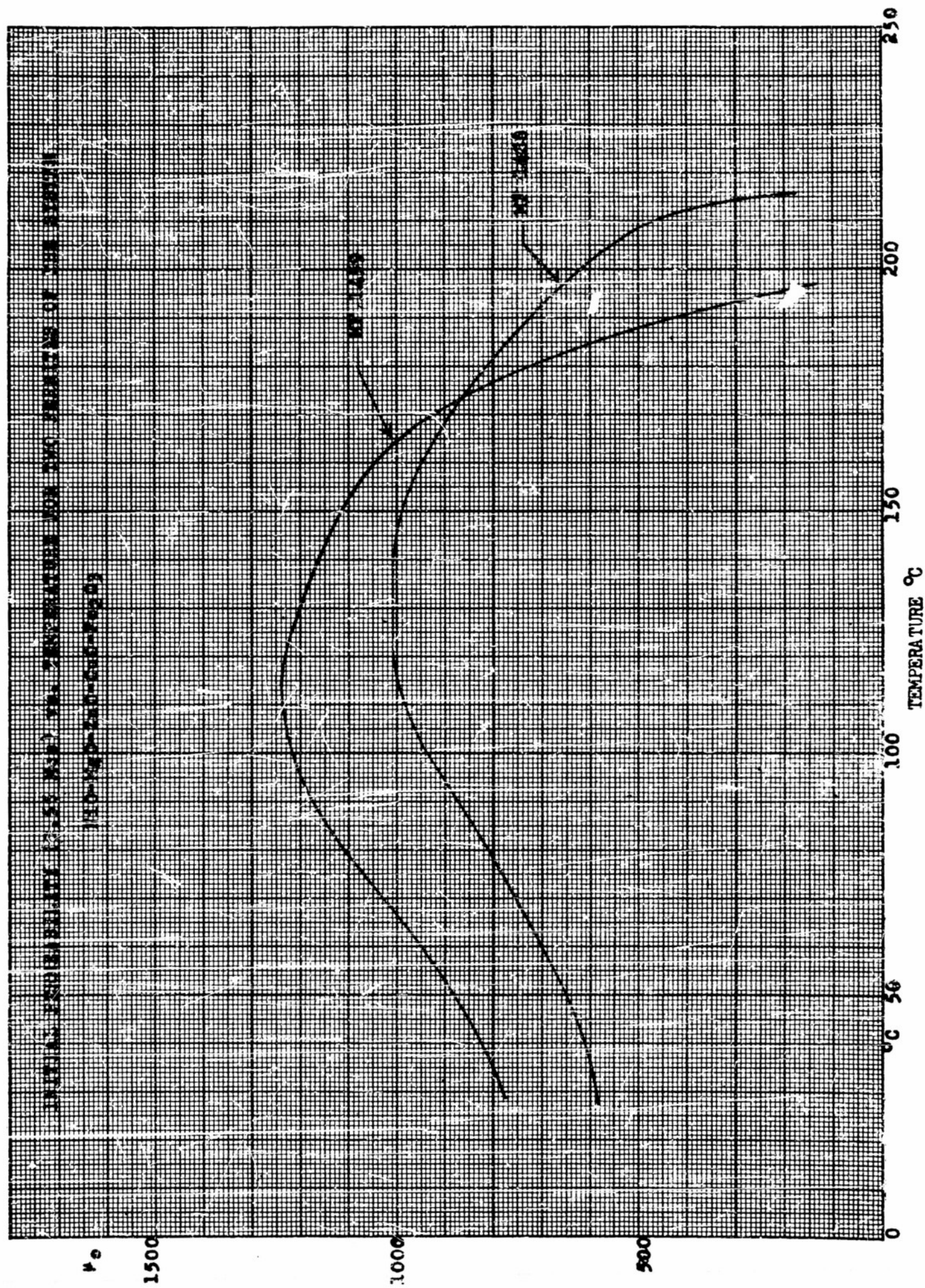| PRESSING PRESSURE	THROUGH 20 MESH		THROUGH 20 MESH ON 80 MESH	
	WITHIN ± 2-1/2%	WITHIN ± 5%	WITHIN ± 2-1/2%	WITHIN ± 5%
3120 PSI	86.0%	100%	88.6%	100%
2080 PSI	66.0%	95.5%	84.0%	100%
1040 PSI	59.0%	91.0%	70.5%	97.8%

GENERAL CERAMICS & STEATITE CORPORATION

GRAPH 167   Sept. 29 - 1953

INITIAL PERMEABILITY (CLASS N3) VS. TEMPERATURE FOR THE CENTER OF THE RANGE

110-140-240-25D

1500

1000

500

50   100   150   200   250

TEMPERATURE °C

SATURATED HYSTERESIS LOOPS (60 cps) FOR THREE FERRITES COVERING A
WIDE RANGE OF NICKEL OXIDE CONTENT
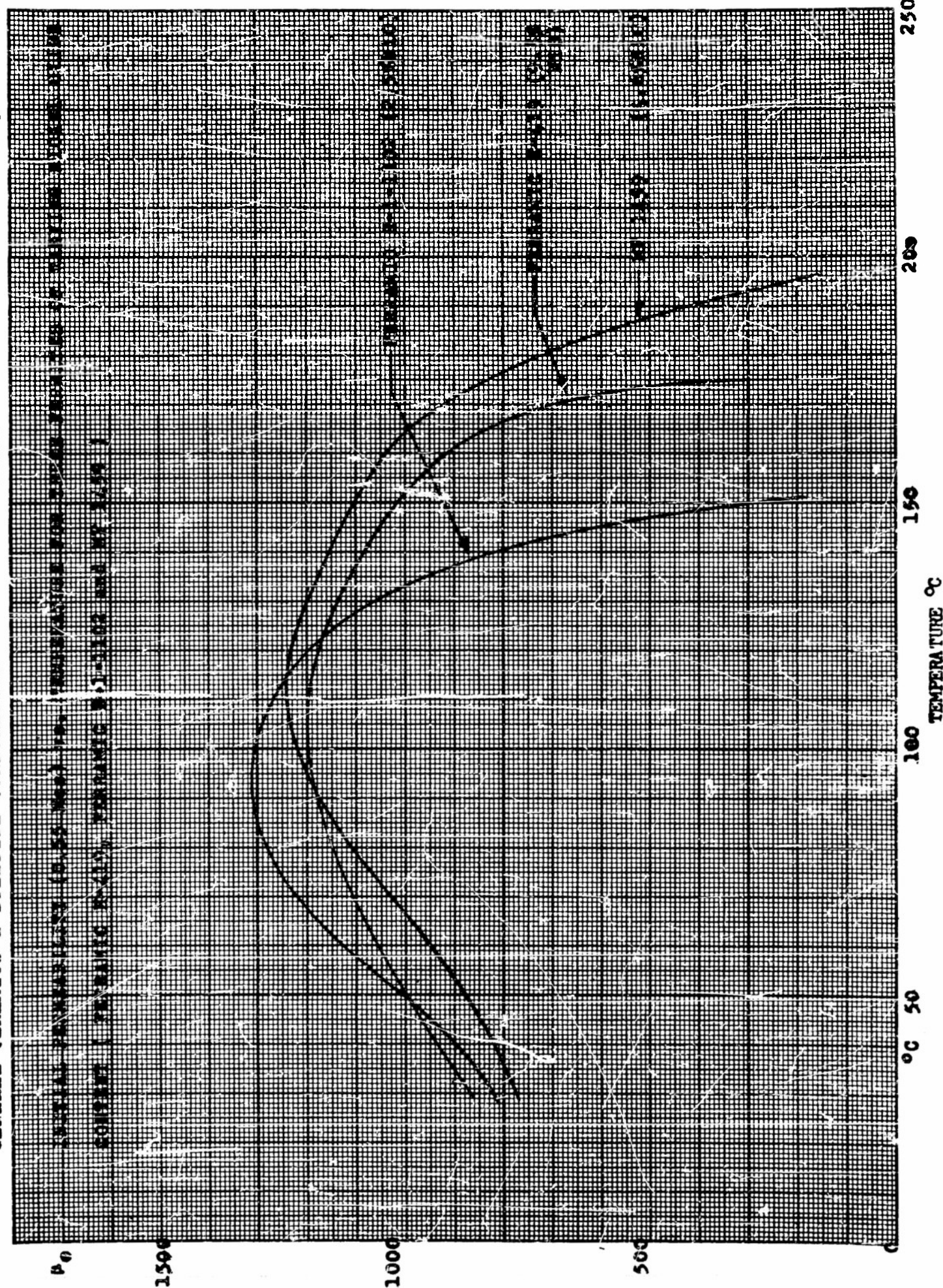

H-1 Body (MF 1102)
Oxide system $MgO-NiO-ZnO-CuO-Fe_2O_3$
Nickel Content: 2.5 %
Magnetic Specifications:
B_s = 2800 gauss
H_c = 0.35 oersteds (maximum)
μ_{max} = 3800

MF 1459
Oxide System $MgO-NiO-ZnO-CuO-Fe_2O_3$
Nickel Content: 4.6 %
Magnetic Specifications:
B_s = 2860 gauss
H_c = 0.19 oersteds
μ_{max} = 3500

H-Body (MF 419)
Oxide System $NiO-ZnO-CuO-Fe_2O_3$
Nickel Content 9.5 %
Magnetic Specifications:
B_s = 3400 gauss
H_c = 0.18 oersteds
μ_{max} = 4300

B₀

1500

1000

500

0

°C   50   100   150   200   250

TEMPERATURE °C

DISTRIBUTION OF Z_0 (LOG) OF MF 254 BODY AT 2400°C, 2 FIRINGS. TWO DIFFERENT FIRINGS, SIEVED THRICE DIFFERENT FRACTIONS. (BIB B-204)

GENERAL CERAMICS & STEATITE CORPORATION   GRAPH Nr. 171   10-20-1953